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A Rapid Method for the

Determination of the Metals of the

R_2O_3 Precipitate in Silicate Analysis

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**A RAPID METHOD FOR THE DETERMINATION
OF THE
METALS OF THE R_2O_3 PRECIPITATE
IN SILICATE ANALYSIS**

BY

ALFRED MARTIN HEINZELMANN

THESIS

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THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

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of the Metals of the R_2O_3 Precipitate in Silicate Analysis*

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A RAPID METHOD FOR THE DETERMINATION OF THE METALS
OF THE
 R_2O_3 PRECIPITATE IN SILICATE ANALYSIS.

The difficulties encountered in the analysis of siliceous rocks are very great, due to the complicated methods of separation which have been necessary for accurate work. The various methods for the separation of the metals of the ammonium sulphide group are all very tedious, requiring repeated precipitations and filtrations to satisfy the needs of quantitative determinations.

Titanium and iron may be estimated in the presence of one another by reducing the latter in slightly acid solution with hydrogen sulphide.* The resulting solution is divided

*Analytical Chemistry by Treadwell-Hall, volume 2 page 114.

into two portions, and the reduced iron of one titrated with permanganate, while the unreduced titanium of the other is precipitated with sodium acetate. However, if iron is present in any quantity, it will always contaminate the TiO_2 precipitate, and a fusion of the mixture is necessary to remove it completely. Also, the method is not applicable if aluminium is present.

The three metals (Fe, Ti, Al) may be estimated in the same sample by the method of Gooch.* This consists in

*Chemical News, 52:55 & 68.

removing the iron as the sulfide in the presence of tartaric acid, then destroying the tartaric acid with permanganate and precipitating the titanium by boiling the solution with ammonium acetate. However, the titanous oxide obtained is always contaminated with the oxides of aluminium and manganese, and must be fused two or three successive times with sodium carbonate for complete removal of these impurities. The aluminium oxide is then gotten by difference.

Walker's method* for separating titanium from iron is

*Percy H. Walker, J. Am. Chem. Soc. 20:513 (1898)

based upon the fact that hydrogen peroxide does not prevent the precipitation of ferric hydroxide by ammonia, but a solution of a titanous salt gives with the same treatment a yellow solution, which if kept cold does not precipitate at once with ammonia. Experiments have shown that a single precipitation is far from giving a complete separation, it being necessary to dissolve the ferric hydroxide in hydrochloric acid, and then to treat with ammonia and hydrogen peroxide, at least three times, in order to obtain results that are quantitative.

According to Baskerville,* titanium may be precipitated

*Chas. Baskerville, J. Am. Chem. Soc. 16:427 (1894)

in the presence of iron and aluminium by boiling a solution of the chlorides with sulfurous acid. The statement is made that a single precipitation is quantitative for the titanium, and

that it is not contaminated with iron. The data given does not support this claim, and later work has shown that the method is unsatisfactory.

The conclusion drawn is that gravimetric separations are either inaccurate or very tedious, and that for ordinary purposes, a reasonably accurate volumetric method for the analysis of the R_2O_3 precipitate is to be desired. Titanium and iron have each been determined by different volumetric methods with varying degrees of success.

Wells and Mitchell* have determined titanium and iron in

*The Volumetric Determination of Titanic Acid and Iron in Ores.
H. L. Wells and W. L. Mitchell, J. Am. Chem. Soc. 17:879.

the presence of one another by treating sulfuric acid solutions of the two metals with hydrogen sulphide. This has no effect upon the titanium, but the iron is reduced to the ferrous condition, so that it may be titrated with standard potassium permanganate. The resulting solution is then reduced with zinc, and again titrated with permanganate, the titanium being obtained by the difference in the two titrations. The results which the authors obtained for titanium are, however, all quite low.

Methylene blue* has also been used as an oxidizing agent in

*Titration of Titanium by Means of Methylene Blue.
B. Neuman and R. K. Murphy, Zeit. angew. Chem. 26:613 (1913)

the determination of titanium, and the results obtained are quite satisfactory. However, the end point of the titration

is attained rather slowly, and certain conditions must be exactly observed. Also, the solution of methylene blue requires frequent standardization.

Knecht and Hibbert,* after studying the very strong reducing

*Titanous Chloride in Volumetric Analysis.

Edmund Knecht and Eva Hibbert, Ber. 36:1549 (1903)

action of titanous chloride, used this as a reagent in titrating for iron. The reaction taking place is represented by the equation:



and is based upon the fact, discovered by Knecht, that titanous chloride reduces ferric salts immediately in the cold. Using potassium thiocyanate as indicator, very satisfactory results have been obtained.

More recently, Ball and Smith* have determined titanium by

*The Volumetric Estimation of Titanium by Means of Ferric Chloride.

T. R. Ball and G. McP. Smith, J. Am. Chem. Soc. 36:1838 (1914)

the same method, in an atmosphere of carbon dioxide to prevent the oxidation of the titanous salt by the air. The results obtained in this way are very exact.

Since both of these determinations have proved to be successful, it was decided to attempt the determination of iron and titanium in the same sample. Or if the mixed oxides were first weighed, aluminium might also be determined by difference, as is now done in most analyses.

The following scheme of analysis was therefore adopted:

The acid solution, containing titanium, aluminium, and iron, is treated with ammonia, and the precipitate filtered ignited, and weighed. The mixture of the oxides is then fused with potassium pyrosulfate and a little potassium fluorid, and the mass is dissolved in dilute hydrochloric acid. The acid solution is reduced with zinc and immediately titrated with standard ferric chloride solution. This gives directly the amount of titanium. The ferrous iron is then oxidized by means of hydrogen peroxide and titrated with standard titanous chloride solution. Knowing the amount of iron added in the previous titration, the amount present at the start may be calculated. The changes involved in the analysis are shown in the following table:

FeCl_3	TiCl_4	$\text{Al}_2(\text{SO}_4)_3$	
↓	↓	↓	precipitate with ammonia, ignite to oxide, and weigh.
Fe_2O_3	TiO_2	Al_2O_3	
↓	↓	↓	fuse with $\text{K}_2\text{S}_2\text{O}_7$, and KF , and dissolve in HCl .
FeCl_3	TiCl_4	AlCl_3	reduce with zinc.
↓	↓	↓	titrate with ferric chloride.
FeCl_2	TiCl_3	AlCl_3	
↓	↓	↓	
FeCl_2	TiCl_4	AlCl_3	* FeCl_2
↓	↓	↓	oxidize with H_2O_2 .
FeCl_3	TiCl_4	AlCl_3	↓
↓	↓	↓	titrate with TiCl_3 .
FeCl_2	TiCl_4	AlCl_3	↓
			FeCl_2

* from FeCl_3 added in titration.

EXPERIMENTAL.

EXPERIMENTAL.

In each analysis, known amounts of the metals were used in solutions made up and standardized as follows:

Titanic Sulphate Solution, $\text{Ti}(\text{SO}_4)_2$.

molecular weight K_2TiF_6	-----	240.3
molecular weight TiO_2	-----	80.1

$$\frac{240.3 \times 5}{80.1} = 15 \text{ grams } \text{K}_2\text{TiF}_6 \text{ to furnish 5 grams } \text{TiO}_2.$$

Exactly 15 grams of potassium fluotitanate, K_2TiF_6 , were evaporated three times to white fumes of SO_3 in a platinum dish with sulfuric acid. An attempt was made to dissolve the residue in 5 percent sulphuric acid, but a small amount of a white powder remained undissolved. The concentration of the acid was increased to about 10 percent and heated, which resulted in the complete solution of the titanium. The final volume of the solution was one liter. This was then standardized by precipitating 10 cubic centimeter portions with ammonia, filtering, and igniting to titanic oxide, weights as follows:

8.7902 gm	weight of crucibles	8.4346 gm
8.8400 gm	weight of crucibles plus TiO_2	8.4847 gm
<u>.0498 gm</u>	weight of TiO_2	<u>.0501 gm</u>

8.4742 gm	weight of crucibles	7.7458 gm
8.5243 gm	weight of crucibles plus TiO_2	7.7957 gm
<u>.0501 gm</u>	weight of TiO_2	<u>.0499 gm</u>

mean value, .0500 gm TiO_2 per 10 cc.

Ferric Chloride Solution, FeCl_3 .

molecular weight FeCl_3	-----	162.23
molecular weight Fe_2O_3	-----	159.70

$$\frac{162.23 \times 2 \times 10}{159.7} = 23.16 \text{ gm FeCl}_3 \text{ to furnish 10 gm Fe}_2\text{O}_3.$$

About 20 grams of ferric chloride were dissolved in water and diluted to one liter. This was standardized gravimetrically by precipitating 10 cubic centimeter portions with ammonia, filtering, and igniting to the oxide, weights as follows:

7.8202 gm	weights of crucibles	8.1109 gm
<u>7.8893 gm</u>	weights of crucibles plus Fe ₂ O ₃	<u>8.1800 gm</u>
.0691 gm	weights of Fe ₂ O ₃	.0691 gm

As a check on the gravimetric method, the iron was also determined by the permanganate titration, i.e. 10 cc. of the ferric chloride solution were evaporated with sulfuric acid to fumes of sulfur trioxide, diluted, passed thru a Jones' Reductor, and titrated with .1236 N. KMnO₄.

0.00	First reading of burettes	7.69
<u>7.03</u>	Second reading of burettes	<u>14.76</u>
7.03	cc. KMnO ₄ required	7.07

$$1 \text{ cc. } .1236 \text{ N. KMnO}_4 = .009869 \text{ gm Fe}_2\text{O}_3.$$

.0694	gm Fe ₂ O ₃ per 10 cc. of solution	.0698
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The mean value of the gravimetric determinations, .0691 gm per 10 cc. was considered as correct.

Potassium Aluminium Sulphate, KAl(SO₄)₂.12H₂O.

molecular weight alum	-----	474.6
molecular weight Al ₂ O ₃	-----	102.2

$$\frac{474.6 \times 2 \times 10}{102.2} = 92.87 \text{ gm KAl(SO}_4)_2.12\text{H}_2\text{O, to furnish 10 gm Al}_2\text{O}_3.$$

93 grams of pure potassium aluminium sulfate were dissolved in water, and diluted to one liter. This was then standardized

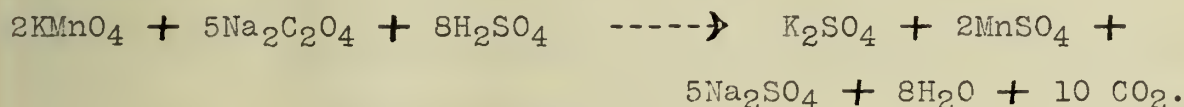
by precipitating 10 cubic centimeter portions with ammonium hydroxide, filtering, and igniting to constant weight over the blast lamp, weights as follows:

8.4847 gm	weights of crucibles	8.5243 gm
8.5855 gm	weights of crucibles plus Al_2O_3	8.6256 gm
<u>.1008 gm</u>	weights of Al_2O_3	<u>.1013 gm</u>

mean value = .1010 gm of Al_2O_3 per 10 cc. of solution.

Potassium Permanganate, KMnO_4 .

The permanganate used instandardizing the ferric chloride solution had been standing about a year. It was filtered thru glass wool and asbestos, and then standardized against pure sodium oxalate. The following represents the equation involved:



$$\frac{2\text{KMnO}_4}{5\text{Na}_2\text{C}_2\text{O}_4} = \frac{316.06}{670} = \frac{x}{\text{gm Na}_2\text{C}_2\text{O}_4}$$

$$x = \frac{316.06 \times \text{gm Na}_2\text{C}_2\text{O}_4}{670} = \text{gm KMnO}_4.$$

$$\frac{316.06 \times \text{gm Na}_2\text{C}_2\text{O}_4 \times 1000}{670 \times 31.606 \times \text{no. cc.}} = \text{N. F.}$$

18.6694 gm	weight of tube plus $\text{Na}_2\text{C}_2\text{O}_4$	18.4290 gm
18.4290 gm	weight of tube	18.1941 gm
<u>.2404 gm</u>	weight of $\text{Na}_2\text{C}_2\text{O}_4$	<u>.2349 gm</u>

0.00	first reading of burettes	0.00
28.95	second reading of burettes	28.33
<u>28.95</u>	cc. KMnO_4 required	<u>28.33</u>

.12398	normality factor of KMnO_4	.12375
--------	-------------------------------------	--------

18.1949 gm	weight of tube plus $\text{Na}_2\text{C}_2\text{O}_4$	17.9592 gm
<u>17.9592 gm</u>	weight of tube	<u>17.6924 gm</u>
.2357 gm	weight of $\text{Na}_2\text{C}_2\text{O}_4$.2668 gm

0.00	first reading of burettes	0.00
<u>28.51</u>	second reading of burettes	<u>32.25</u>
28.51	cc. KMnO_4 required	32.25

.12339	normality factor of KMnO_4	.12348
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average N. F. of four determinations -- .1236

Ferric Chloride Titrating Solution, FeCl_3 .

About two liters of approximately tenth normal ferric chloride solution were made up for use in titrating against titanous salts. As in the previous instances, this was standardized by precipitation with ammonia, and ignition to ferric oxide. 20 cubic centimeters were used for this purpose.

8.7911 gm	weight of crucibles	7.7466 gm
<u>8.9225 gm</u>	weight of crucibles plus Fe_2O_3	<u>7.8786 gm</u>
.1314 gm	weight of Fe_2O_3	.1320 gm

$$\frac{\text{gm } \text{Fe}_2\text{O}_3 \times 1000}{20 \times 79.84} = \text{normality factor.}$$

$$\frac{.1317 \times 1000}{20 \times 79.84} = .0825 \text{ the N. F. of } \text{FeCl}_3.$$

Titanous Chloride Solution, TiCl_3 .

The titanous chloride solution used in titrating for iron was made by diluting Kahlbaum's 15 percent solution with about six or seven times its volume of freshly boiled distilled water. The reagent was preserved out of contact with the air in a two liter bottle connected with a carbon dioxide generator, and with a burette. It was standardized by titration against the ferric chloride solution every time it

was used. The following is the result of the first titration made just preceeding the first analysis, 1 1/2 cc. saturated solution of potassium thiocyanate being used as indicator. The titanous chloride was added to the ferric chloride so as to prevent oxidation of the former by air, since titanous salts are oxidized much more rapidly than ferrous.

FeCl ₃		TiCl ₃
.78	first reading of burettes	1.76
<u>25.86</u>	second reading of burettes	<u>27.51</u>
25.08	cc. required	25.75

$$\frac{25.75}{25.08} = 1.0267, \text{ the ratio.}$$

.93	first reading of burettes	1.67
<u>26.01</u>	second reading of burettes	<u>27.42</u>
25.08	cc. required	25.75

$$\frac{25.75}{25.08} = 1.0267, \text{ the ratio.}$$

.90	first reading of burettes	1.61
<u>25.70</u>	second reading of burettes	<u>27.06</u>
24.80	cc. required	25.45

$$\frac{25.45}{24.80} = 1.0262, \text{ the ratio.}$$

mean ratio from three determinations equals 1.0265

$$\frac{.0825}{1.0265} = .0804, \text{ the N. F. of the TiCl}_3.$$

ANALYSIS OF SAMPLE #1

Sample #1 was made up of the following composition:

10 cc. FeCl ₃	or	.0691 gm Fe ₂ O ₃
10 cc. TiCl ₄	or	.0500 gm TiO ₂
10 cc. KAl(SO ₄) ₂	or	.1010 gm Al ₂ O ₃

The sample represented above was diluted with 250 cc. of water,

7 grams of ammonium nitrate were added, to keep the aluminium hydroxide in the hydrogel form, and the solution heated to boiling. A slight excess of ammonia was added, the precipitate allowed to settle, filtered, washed, and ignited strongly for one half hour.

20.4690 gm	weight of crucible	20.4881 gm
<u>20.6890 gm</u>	weight of crucible plus oxides	<u>20.7078 gm</u>
.2200 gm	weight of oxides	.2197 gm

The mixed oxides were then fused in a platinum crucible with .5 gm of potassium fluoride and 2.5 gm of potassium pyrosulfate, and the fused mass dissolved in hot dilute hydrochloric acid. The solution was then reduced with 20 mesh zinc in a separatory funnel as described by Ball and Smith*, transferred to a flask

*The Volumetric Estimation of Titanium by Means of Ferric Chloride.
T. R. Ball and G. McP. Smith, J. Am. Chem. Soc. 36:1841 (1914)

filled with carbon dioxide, and immediately titrated with ferric chloride solution.

0.00	first reading of burettes	0.00
<u>7.67</u>	second reading of burettes	<u>7.63</u>
7.67	cc. FeCl ₃ required	7.63

The solution was then oxidized with hydrogen peroxide, heated to boiling to decompose the excess of hydrogen peroxide, cooled to room temperature, and titrated with TiCl₃.

4.55	first reading of burettes	6.55
<u>17.22</u>	second reading of burettes	<u>18.95</u>
12.67	cc. TiCl ₃ used	12.40

Results from Sample #1

TiO ₂		Fe ₂ O ₃		Al ₂ O ₃	
found	calculated	found	calculated	found	calculated
.0507	.0500	.0617	.0691	.1076	.1010
.0504	.0500	.0587	.0691	.1106	.1010

ANALYSIS OF SAMPLE #2

Sample #2 was made up of the following composition:

10 cc. FeCl_3	or	.0691 gm Fe_2O_3
20 cc. $\text{Ti}(\text{SO}_4)_2$	or	.1000 gm TiO_2
5 cc. $\text{KAl}(\text{SO}_4)_2$	or	.0505 gm Al_2O_3

The ratio between FeCl_3 and TiCl_3 was determined and found to be 1.4786*. The sample was then analysed as in the previous

*The reason for the great increase in this ratio was due to a defective burette which was replaced after the analysis of Sample #1.

case as follows:

20.4685 gm	weight of crucible	20.4875 gm
20.6886 gm	weight of crucible plus oxides	20.7079 gm
<u>.2201 gm</u>	weight of oxides	<u>.2204 gm</u>
0.00	first reading of burettes	0.00
15.21	second reading of burettes	15.17
<u>15.21</u>	cc. FeCl_3 required	<u>15.17</u>
8.75	first reading of burettes	8.83
29.85	second reading of burettes	26.12
<u>21.10</u>	cc. TiCl_3 required	<u>17.29</u>

Results from Sample #2

TiO_2		Fe_2O_3		Al_2O_3	
found	calculated	found	calculated	found	calculated
.1005	.1000	-.0060	.0691	.1256	.0505
.1003	.1000	-.0231	.0691	.1432	.0505

ANALYSIS OF SAMPLE #3

Sample #3 was made up of the following composition:

10 cc. FeCl_3	or	.0691 gm Fe_2O_3
20 cc. $\text{Ti}(\text{SO}_4)_2$	or	.1000 gm TiO_2
5 cc. $\text{KAl}(\text{SO}_4)_2$	or	.0505 gm Al_2O_3

20.4678 gm	weight of crucible	20.4869 gm
20.6860 gm	weight of crucible plus oxides	20.7072 gm
<u>.2198 gm</u>	weight of oxides	<u>.2203 gm</u>
0.00	first reading of burettes	0.00
15.12	second reading of burettes	15.07
<u>15.12</u>	cc. FeCl ₃ required	<u>15.07</u>
9.91	first reading of burettes	12.42
29.86	second reading of burettes	29.90
<u>19.95</u>	difference	<u>17.48</u>
9.86	third reading of burettes	10.14
13.86	fourth reading of burettes	15.09
<u>23.95</u>	cc. TiCl ₃ required	<u>22.43</u>

Results from Sample #3

TiO ₂		Fe ₂ O ₃		Al ₂ O ₃	
found	calculated	found	calculated	found	calculated
.0999	.1000	.0072	.0691	.1127	.0505
.0997	.1000	.0006	.0691	.1200	.0505

ANALYSIS OF SAMPLE #4

Sample #4 was made up of the following composition:

20 cc. FeCl ₃	or	.1382 gm Fe ₂ O ₃
10 cc. Ti(SO ₄) ₂	or	.0500 gm TiO ₂
5 cc. KAl(SO ₄) ₂	or	.0505 gm Al ₂ O ₃

The TiCl₃ was again titrated against the FeCl₃, and the ratio 1.4885 found.

20.4671 gm	weight of crucibles	20.4859 gm
20.7056 gm	weight of crucibles plus oxides	20.7250 gm
<u>.2385 gm</u>	weight of oxides	<u>.2391 gm</u>
0.00	first reading of burettes	0.00
7.54	second reading of burettes	7.50
<u>7.54</u>	cc. FeCl ₃ required	<u>7.50</u>
9.80	first reading of burettes	9.79
13.05	second reading of burettes	23.10
<u>3.25</u>	cc. TiCl ₃ required	<u>13.31</u>

Results from Sample #4

TiO ₂		Fe ₂ O ₃		Al ₂ O ₃	
found	calculated	found	calculated	found	calculated
.0498	.0500	-.0353	.1382	.2240	.0505
.0496	.0500	.0046	.1382	.1849	.0505

The complete results obtained by this method may be tabulated as follows:

TiO ₂		Fe ₂ O ₃		Al ₂ O ₃	
found	calculated	found	calculated	found	calculated
.0507	.0500	.0617	.0691	.1076	.1010
.0504	.0500	.0587	.0691	.1106	.1010
.1005	.1000	-.0060	.0691	.1256	.0505
.1003	.1000	-.0231	.0691	.1432	.0505
.0999	.1000	.0072	.0691	.1127	.0505
.0997	.1000	.0006	.0691	.1200	.0505
.0498	.0500	-.0353	.1382	.2240	.0505
.0496	.0500	.0046	.1382	.1849	.0505

It will be observed that the titanium checks very well with the calculated values thruout, but that the iron and therefore also the aluminium do not check even in the slightest degree with the results of the gravimetric determinations. The error of the method must clearly have been introduced during the titration with ferric chloride. It was noticed that after the titration of the ferric iron with the titanous salt, the solution had a decided turbidity, due to colloidal sulfur, and also a perceptible odor of sulfur dioxide. From this fact, and also because the values for iron were all low, the reason for the failure of the method was not far to seek. In the oxidation with hydrogen peroxide, in acid solution, the potassium

thiocyanate was decomposed, giving free sulfur, sulfur dioxide, and perhaps other products. The sulfur dioxide, being a strong reducing agent, would reduce part of the ferric iron, and thereby decrease the quantity of titanous chloride required in the titration. To obviate this difficulty, the method was modified as follows:

The acid solution of titanium, iron, and aluminium is treated with ammonia, and the precipitate filtered, ignited, and weighed. This is then fused as before with potassium pyrosulfate and potassium fluoride, the mass being dissolved in dilute hydrochloric acid. The solution is then divided into two equal portions. One is titrated directly with titanous chloride, while the other is reduced with zinc, and titrated with ferric iron. This method proved to be very successful, as is shown by the following results:

ANALYSIS OF SAMPLE #5

Sample #5 was made up of the following composition:

10 cc. $\text{Ti}(\text{SO}_4)_2$	or	.0500 gm TiO_2
20 cc. FeCl_3	or	.1382 gm Fe_2O_3
5 cc. $\text{KAl}(\text{SO}_4)_2$	or	.0505 gm Al_2O_3

20.4678 gm	weight of crucibles	20.4865 gm
20.7063 gm	weight of crucibles plus oxides	20.7256 gm
<u>.2385 gm</u>	weight of oxides	<u>.2391 gm</u>
0.00	first reading of burettes	0.00
3.75	second reading of burettes	3.79
<u>3.75</u>	cc. FeCl_3 required	<u>3.79</u>

10.02	first reading of burettes	11.33
<u>25.76</u>	second reading of burettes	<u>27.13</u>
15.74	cc. TiCl_3 required	15.80

Results from Sample #5

Fe_2O_3		TiO_2		Al_2O_3	
found	calculated	found	calculated	found	calculated
.1386	.1382	.0496	.0500	.0503	.0505
.1390	.1382	.0501	.0500	.0500	.0505

ANALYSIS OF SAMPLE #6

Sample #6 was made up of the following composition:

10 cc. FeCl_3	or	.0691 gm Fe_2O_3
20 cc. $\text{Ti}(\text{SO}_4)_2$	or	.1000 gm TiO_2
5 cc. $\text{KAl}(\text{SO}_4)_2$	or	.0505 gm Al_2O_3

20.4670 gm	weight of crucibles	20.4857 gm
<u>20.6869 gm</u>	weight of crucibles plus oxides	<u>20.7061 gm</u>
.2199 gm	weight of oxides	.2204 gm

0.00	first reading of burettes	0.20
<u>7.54</u>	second reading of burettes	<u>7.80</u>
7.54	cc. FeCl_3 required	7.60

11.47	first reading of burettes	11.65
<u>19.36</u>	second reading of burettes	<u>19.47</u>
7.89	cc. TiCl_3 required	7.82

Results from Sample #6

Fe_2O_3		TiO_2		Al_2O_3	
found	calculated	found	calculated	found	calculated
.0694	.0691	.0997	.1000	.0508	.0505
.0688	.0691	.1004	.1000	.0512	.0505

ANALYSIS OF SAMPLE #7

Sample #7 was made up of the following composition:

10. cc. FeCl_3	or	.0691 gm Fe_2O_3
10. cc. $\text{Ti}(\text{SO}_4)_2$	or	.0500 gm TiO_2
5. cc. $\text{KAl}(\text{SO}_4)_2$	or	.0505 gm Al_2O_3

20.4668 gm	weight of crucibles	20.4855 gm
20.6360 gm	weight of crucibles plus oxides	20.6554 gm
<u>.1692 gm</u>	weight of oxides	<u>.1699 gm</u>

0.00	first reading of burettes	3.73
3.73	second reading of burettes	<u>7.50</u>
<u>3.73</u>	cc. FeCl_3 required	<u>3.77</u>

12.47	first reading of burettes	11.90
20.34	second reading of burettes	<u>19.82</u>
<u>7.87</u>	cc. TiCl_3 required	<u>7.92</u>

Results from Sample #7

Fe_2O_3		TiO_2		Al_2O_3	
found	calculated	found	calculated	found	calculated
.0693	.0691	.0493	.0500	.0510	.0505
.0696	.0691	.0498	.0500	.0505	.0505

The ratio between the ferric chloride and titanous chloride during the last three analyses was 1.4975. The complete results from these three determinations may be tabulated as follows:

Fe_2O_3		TiO_2		Al_2O_3	
found	calculated	found	calculated	found	calculated
.1386	.1382	.0496	.0500	.0503	.0505
.1390	.1382	.0501	.0500	.0500	.0505
.0694	.0691	.0997	.1000	.0508	.0505
.0688	.0691	.1004	.1000	.0512	.0505
.0693	.0691	.0493	.0500	.0510	.0505
.0696	.0691	.0498	.0500	.0505	.0505

CONCLUSIONS.

It is seen that this method gives very good results, and is certainly much shorter than the tedious gravimetric separations used at the present time. The precipitation and filtration of the mixed oxides requires the most time, but this operation is necessary in any determination. The reduction with zinc was allowed to proceed for one and one half hours in each case.

CONCLUSIONS

Iron, aluminium, and titanium may be determined in the presence of one another by weighing their combined oxides, and then after dissolving the mixture, titrating one half for iron with titanous chloride and the other half for titanium with ferric chloride.

The method is both rapid and accurate.

The titration for iron by means of titanous chloride eliminates the operation of reduction, which is necessary in the permanganate or dichromate titrations.

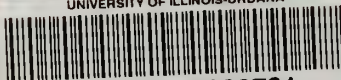
The direct titration for titanium in the presence of iron eliminates the very tedious separations necessary in gravimetric determinations.

In the presence of vanadium and other similar metals gravimetric methods would of course have to be used.





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